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Description

Specification

Process for the preparation of organically modified, permanently hydrophobic aerogels

The invention pertains to a process for the preparation of organically modified, permanently hydrophobic aerogels.

Aerogels, especially those with porosities above 60% and densities below 0.6 g/cm^3, have an extremely low thermal conductivity and therefore find use as a thermal insulation material as described, for example, in EP-A-0 171 722.

Aerogels in the wider sense, i.e. in the sense of a "gel with air as the dispersing agent," are prepared by drying a suitable gel. Aerogels in the narrower sense, xerogels and cryogels, are included in the concept of an "aerogel" in this sense. In this connection, a dried gel is termed an aerogel in the narrower sense if the liquid of the gel is removed at temperatures above the critical temperature and starting out from pressures above the critical pressure. If, by contrast, the liquid of the gel is removed subcritically, e.g. with the formation of a liquid/vapor boundary phase, then one designates the produced gel a xerogel.

When using the term aerogels in the present application, one is dealing with aerogels in the wider sense, i.e. in the sense of a "gel with air as the dispersing agent."

In addition, one can basically subdivide aerogels into inorganic and organic aerogels.

Inorganic aerogels have been known since 1931 (S.S. Kistler, Nature 1931, 127, 741). Since then, aerogels have been produced from the most varied initial materials. For example, SiO_2, Al_2O_3, TiO_2, ZrO_2, SnO_2, Li_2O, CeO_2 and V_2O_5 aerogels as well as mixtures of these could be prepared (H. D. Gesser, P. C. Goswami, Chem. Rev. 1989, 89, 765 ff).

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~~For several years, organic aerogels have also been known. One finds in the literature, e.g., organic aerogels based on resorcinol/formaldehyde, melamine/formaldehyde or resorcinol/furfurol (R. W. Pekala, J. Mater. Sci. 1989, 24, 3221, US-A 5,508,341, RD 388,047 [368,047?], WO94/22,943 and US-A-5,556,892). In addition, organic aerogels of polyisocyanates (WO95/03,358) and polyurethanes (US-5,484,818) are also known. One proceeds from initial materials such as formaldehyde and resorcinol dissolved in water, as described, for example, in US-A-5,508,341; these are brought to reaction with one another by suitable catalysts, the water in the pores of the gel that forms is exchanged for a suitable organic solvent, and then the gel is dried supercritically.~~

Inorganic aerogels can be prepared in different ways.

First of all, SiO_2 aerogels can be produced by acid hydrolysis and condensation of tetraethylorthosilicate in ethanol. A gel is thus formed, which can be dried by supercritical drying while retaining its structure. Production processes based on this drying technique are known, e.g., from EP-A-0 396,076; WO 92/03,378; and WO 95/06,617.

An alternative to the above drying is offered by a process for subcritical drying of SiO_2 gels, in which these are reacted with a silylation agent containing

chlorine prior to drying. The SiO_2 gel can be obtained, for example, by acid hydrolysis of tetraalkoxysilanes, preferably tetraethoxysilanes (TEOS), in a suitable organic solvent, preferably ethanol, by means of water. After exchange of the solvent for a suitable organic solvent, the obtained gel is reacted with a chlorine-containing silylation agent in an additional step. Methylchlorosilanes ($Me_{4-n}SiCl_n$ with n = 1 to 3) are thus preferably used as silylation agents, due to their reactivity. The SiO_2 gel that is thus formed and is modified with methylsilyl groups on its surface can then be dried in air from an organic solvent. Thus aerogels with densities below 0.4 g/cm^3 and porosities greater than 60% can be obtained. The production process based on this drying technique is described in detail in WO 94/25,149.

The above-described gels can be reacted with tetraalkoxysilanes in aqueous alcohol solution prior to drying and can be aged in order to increase the gel network strength, as disclosed, e.g., in WO 92/20,623.

The tetraalkoxysilanes used in the above-described process as initial materials, however, represent an extraordinarily high cost factor.

A considerable reduction in cost can be achieved by the use of water glass as the initial material for the production of SiO_2 gels. For this purpose, for example, a silicic acid can be produced from an aqueous water-glass solution by means of an ion-exchange resin, and this acid can be polycondensed by addition of a base to form a SiO_2 gel. After exchange of the aqueous medium for a suitable organic solvent, the obtained gel is then converted with a chlorine-containing silylation agent in an additional step. Methylchlorosilanes ($Me_{4-n}SiCl_n$

with n = 1 to 3) are also preferably utilized as silylation agents, due to their reactivity. The modified SiO_2 gel that is formed and is modified with methylsilyl groups on the surface can then also be dried in air from an organic solvent. The production process based on this technique is described in detail, e.g., in DE-A-4,342,548.

Hydrogen chloride (HCl) as well as a multiple number of byproducts combined therewith are necessarily formed in very large quantities in the silylation by means of chlorine-containing silylation agents, and these require sometimes a very expensive and cost-intensive cleaning of the silylated SiO_2 gels by multiple washings with a suitable organic solvent.

The use of a silylation agent free of chlorine is described in DE-C 195 02,453. For this purpose, for example, a silicate-type lyogel produced according to the above-described method is proposed and is reacted with a chlorine-free silylation agent. Preferably methylisopropene oxysilanes ($Me_{4-n}Si(OC(CH_3)CH_2)_n$ with n = 1 to 3) are used as silylation agents. The thus-formed SiO_2 gel that is modified with methylsilyl groups on the surface can then also be dried in air from an organic solvent.

By the use of chlorine-free silylation agents, in fact, the problem of formation of HCl is solved, but the chlorine-free silylation agents that are used represent a very high cost factor.

In WO 95/06,617 and in the German Patent Application 195-41 279.6, methods are disclosed for the production of silicic acid aerogels with hydrophobic surface groups.

In WO 95/06,617, silicic acid aerogels are obtained by reaction of a water-glass solution with an acid at a pH value of 7.5 to 11, extensive release of the silicic acid hydrogel that forms from the ionic components by washing with water or dilute aqueous solutions of inorganic bases, whereby the pH of the hydrogel is maintained in the range of 7.5 to 11, expelling the aqueous phase obtained in the hydrogel by an alcohol and subsequent supercritical drying of the obtained alkogel.

In German Patent Application 195-41 279.6, in a way similar to that described in WO 95/06,617, silicic-acid aerogels are produced and are then dried subcritically.

In both methods, however, the omission of chlorine-containing silylation agents leads only to an aerogel with hydrophobic surface groups bound via oxygen. These can easily be cleaved again in a water-containing atmosphere. Thus the described aerogel is only hydrophobic for a short time.

It is also possible to utilize organically modified gels without final drying to the aerogel in the most varied fields of technology, such as, e.g., in chromatography, in cosmetics, and in the pharmaceutical field.

The task of the present invention was thus to prepare a method for the production of permanently hydrophobic aerogels, in which a commercially available, inexpensive silylation agent can be used, without incurring the other disadvantages described above, which are known from the prior art.

This task is resolved by a process for the production of organically modified aerogels with permanently hydrophobic surface groups, in which one

a) provides a lyogel,

b) washes the lyogel provided in step a) with an organic solvent,

c) surface-silylates the gel obtained in step b), and

d) dries the surface-silylated gel obtained in step c),

characterized in that in step c), as the silylation agent, a disiloxane of formula I is used

$$R_3Si\text{-}O\text{-}SiR_3 \qquad (I)$$

whereby the residues R, independently of one another, the same or different, indicate in each case a hydrogen atom or a nonreactive organic, linear, branched, cyclic, saturated or unsaturated, aromatic or heteroaromatic residue, preferably C_1-C_{18} alkyl or C_6-C_{14} aryl, and particularly preferred C_1-C_6 alkyl, cyclohexyl or phenyl, particularly methyl or ethyl.

In the present invention, a lyogel is understood to mean a gel dispersed in at least one solvent. The solvent may also be water. If the water component in the solvent amounts to at least 50%, then one also speaks of a hydrogel.

The network of the lyogel may be present in any organic and/or inorganic base composition. All of the systems of the prior art known to the person skilled in the art can be used as the organic base composition. An inorganic base composition is preferably based on oxidic silicon, tin, aluminum, gallium, indium, titanium and/or zirconium compounds, and particularly preferred are those based on oxidic silicon, aluminum, titanium and/or zirconium compounds. Most preferred is a silicate-type hydrogel, which may contain fractions of zirconium, aluminum, titanium, vanadium and/or iron compounds, particularly a pure silicate-

type hydrogel. In the case of organic and/or inorganic base compositions, th different components must not necessarily be distributed homogeneously and/or form a continuous network. It is also possibl that individual components are present partially or completely in the form of inclusions, individual nuclei and/or agglomerations in the network.

The disiloxanes used according to the invention, when compared with the chlorine-containing silylation agents known from the prior art, have the advantage that no chlorine-containing byproducts are formed. In addition, they can easily be separated from aqueous phases based on their insolubility, which makes possible the recovery of excess reagents. In this way, it is possible to minimize silylation times by the use of excess concentrations.

The preparation of the lyogels provided in step a) can be produced according to all methods known to the person skilled in the art.

Three preferred forms of embodiment for the preparation of silicate-type lyogels will be described in more detail in the following, but without, however limitation to these.

In a first preferred form of embodiment, in step a) a silicate-type lyogel is provided, which is obtained by hydrolysis and condensation of Si alkoxides in an organic solvent with water. A tetraalkoxysilane, preferably tetraethoxy- or tetramethoxysilane is used as the Si alkoxide. The organic solvent is thus preferably an alcohol, and particularly preferred ethanol or methanol, to which up to 20 vol.% water can be added. In the hydrolysis and concentration of Si

alkoxides in an organic solvent with water, acids and/or bases may be added in a one- or two-step process as catalysts.

The lyogel provided in step a) may also contain zirconium, aluminum, tin and/or titanium compounds suitable for condensation.

In addition, before and/or during the gel preparation, opacifiers can be added as additives, particularly IR opacifiers for reduction of the radiation contribution to the heat conductivity, such as, e.g., carbon black, titanium oxides, iron oxides, and/or zirconium oxides.

In addition, fibers can be added to the sol in order to increase the mechanical stability. Inorganic fibers, such as, e.g., glass fibers or mineral fibers, organic fibers, such as, e.g., polyester fibers, aramide fibers, nylon fibers or fibers of plant origin, as well as mixtures of these can be used as the fiber materials. The fibers may also be coated, such as, e.g., polyester fibers, which are metallized with a metal, such as, e.g., aluminum.

The production of the lyogel is generally conducted at a temperature between the freezing point of the solution and 70°C. In this way, if necessary, a shaping step can be conducted simultaneously, such as, e.g., spray forming, extrusion or drop formation.

The obtained lyogel can also be subjected to an aging. This is generally done between 20°C and the boiling point of the organic solvent. If necessary, aging can also be conducted under pressure at higher temperatures. The time generally amounts to up to 48 hours, preferably up to 24 hours.

In a second preferred form of embodiment, a silicate-type hydrogel is introduced in step a), which is prepared by bringing an aqueous water-glass solution to a pH of ≤ 3 by means of an acidic ion-exchanger resin, a mineral acid, or a hydrochloric acid solution, then polycondensing the silicic acid that forms thereby by addition of a base to a SiO_2 gel, and, if a mineral acid or a hydrochloric acid solution has been used, the gel is washed with water essentially free of electrolyte. The polycondensation to the SiO_2 gel can be undertaken both in one step as well as in multiple steps.

Preferably sodium and/or potassium water glass are used as the water glass. Preferably an acidic resin is used as the ion-exchanger resin, whereby in particular, those resins are suitable, which contain sulfonic acid groups. If one uses mineral acids, hydrochloric acid and/or sulfuric acid are particularly suitable. If one uses hydrochloric acid solutions, particularly aluminum salts are suitable, especially aluminum sulfate and/or chloride. As the base, generally NH_4OH, NaOH, KOH, $Al(OH)_3$ and/or colloidal silicic acid are used.

The hydrogel preferably prepared from the above-described silicate-type initial compounds may also contain zirconium, aluminum, tin and/or titanium compounds capable of condensation.

In addition, prior to and/or during the gel production, opacifiers may be added as additives, particularly IR opacifiers, for the reduction of the radiation contribution to the heat conductivity, such as, e.g., carbon black, titanium oxides, iron oxides and/or zirconium oxides.

In addition, fibers can be added to the sol for increasing the mechanical stability. Inorganic fibers such as, e.g., glass fib rs or mineral fibers, organic fibers such as e.g., polyester fibers, aramide fibers, nylon fibers or fibers of plant origin, as well as mixtures of the same can be used as fiber materials. The fibers may also be coated, such as, e.g., polyester fibers, which are metallized with a metal, such as, e.g., aluminum.

The production of the hydrogel is generally conducted at a temperature between the freezing point and the boiling point of the solution. Thus, if necessary, a shaping step can be conducted simultaneously, such as, e.g., spray forming, extrusion or drop formation.

The obtained hydrogel can also be subjected to an aging. This aging may be produced prior to and/or after an above-described possible washing with water, with which the gel is essentially washed free of electrolyte.

The aging is conducted generally at a temperature in the range of 20 to 100°C, preferably at 40 to 100°C and particularly at 80 to 100°C, and at a pH value of 4 to 11, preferably 5 to 9, and particularly 5 to 8. The time for this generally amounts to up to 48 hours, preferably up to 24 hours, and particularly preferably up to 3 hours.

In a third preferred form of embodiment, in step a) a silicate-type hydrogel is provided, which is prepared by obtaining a SiO_2 gel from an aqueous water-glass solution by means of at least one organic and/or inorganic acid through the intermediate step of a silicic acid sol.

In general, a 6 to 25 wt.% (with respect to the SiO_2 content) sodium and/or potassium water-glass solution is used as the water-glass solution. Preferred is a 10 to 25 wt.% water-glass solution, and particularly preferred is a 10 to 18 wt.% water-glass solution.

In addition, the water-glass solution may also contain up to 90 wt.%, with respect to the SiO_2, of zirconium, aluminum, tin and/or titanium compounds capable of condensation.

As acids, generally 1 to 50 wt.% acids are used, preferably 1 to 10 wt.% acids. Preferred acids are sulfuric acid, phosphoric acid, hydrofluoric acid, oxalic acid, and/or hydrochloric acid. Particularly preferred is hydrochloric acid. However, mixtures of the corresponding acids may also be utilized.

In addition to the mixing, properly speaking, of the water-glass solution and the acid, it is also possible to introduce a part of the acid into the water-glass solution and/or a part of the water-glass solution into the acid prior to the mixing itself. It is possible in this way to vary the ratio of the material flows of water-glass solution/acid over a very broad range.

After mixing the two solutions, preferably a 5 to 12 wt.% SiO_2 gel is obtained. A 6 to 9 wt.% SiO_2 gel is particularly preferred.

In order to assure an optimal intermixing of the water-glass solution and the acid, before a SiO_2 gel is formed, both solutions should preferably have, independent of one another, a temperature between 0 and 30°C, particularly preferred between 5 and 25°C, and particularly between 10 and 20°C.

The rapid intermixing of the two solutions is conducted in devices known to the person skilled in the art, such as, e.g., boilers with stirring apparatus, mixing nozzles and static mixers. Preferred are semicontinuous or continuous processes, such as, e.g., mixing nozzles.

If necessary, a forming step can be conducted simultaneously with production, e.g., by spray-forming, extrusion or drop formation.

The obtained hydrogel may also be subjected to an aging. This is generally done at 20 to 100°C, preferably at 40 to 100°C, particularly at 80 to 100°C and a pH value of 2.5 to 11, preferably 5 to 8. The time for this generally amounts to up to 12 hours, preferably up to 2 hours, and particularly preferred, up to 30 minutes.

The prepared gel is preferably washed with water, particularly preferably until the wash water used is free of electrolyte. If an aging of the gel is to be conducted, the washing can be conducted prior to, during and/or after the aging, whereby the gel in this case is preferably washed during or after the aging. For the washing, a part of the water can be replaced by organic solvent. The water content, however, should preferably be high enough that the salts in the pores of the hydrogel do not crystallize out.

In order to remove sodium and/or potassium ions as extensively as possible, the hydrogel can also be washed with a mineral salt prior to, during and/or after the washing with water. Preferred mineral salts are thus also the mineral salts named as preferred for the production of the hydrogel.

Further, opacifiers can be added to the water glass, the acid and/ r th sol as additives, particularly IR opacifiers, for the reduction of the radiation contribution to the heat conductivity such as, e.g., carbon black, titanium oxides, iron oxides and/or zirconium oxides.

In addition, fibers can be added to the water glass, to the acid and/or to the sol in order to increase the mechanical stability. Inorganic fibers, such as, e.g., glass fibers or mineral fibers, organic fibers, such as, e.g., polyester fibers, aramide fibers, nylon fibers or fibers of plant origins as well as mixtures of the same can be used as fiber materials. The fibers may also be coated, such as, e.g., polyester fibers, which are metallized with a metal, such as, e.g., aluminum.

In step b), one washes the gel obtained from step a) with an organic solvent, preferably until the water content of the gel is ≤ 5 wt.%, particularly preferred ≤ 2 wt.% and in particular ≤ 1 wt.%. Generally aliphatic alcohols, ethers, esters or ketones as well as aliphatic or aromatic hydrocarbons are used as solvents. Preferred solvents are methanol, ethanol, acetone, tetrahydrofuran, acetic acid ethyl ester, dioxane, pentane, n-hexane, n-heptane and toluene. Particularly preferred as the solvent is acetone, tetrahydrofuran, pentane and n-heptane. Mixtures of the named solvents may also be used. Further, the water can also be washed out first with a water-miscible solvent, e.g., an alcohol, acetone or THF, and then the latter is washed out with a hydrocarbon. Preferably pentane or n-heptane is used as the hydrocarbon.

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The lyogel obtained in step b) may be subjected to an aging. This is generally done between 20°C and the boiling point of the organic solvent. If

necessary, aging may be conducted also under pressure at higher temperatures. The time generally amounts to up to 48 hours, preferably up to 24 hours. After such an aging, if necessary, another solvent exchange for the same or a different solvent can be conducted. This additional aging step may also be repeated several times.

In step c), the solvent-containing gel is reacted with a disiloxane of formula I as the silylation agent.

$$R_3Si\text{-}O\text{-}SiR_3 \qquad (I)$$

whereby the residues R, independently of one another, either the same or different, each time represent a hydrogen atom or a nonreactive organic, linear, branched, cyclic, saturated or unsaturated, aromatic or heteroaromatic residue, preferably C_1-C_{18} alkyl or C_6-C_{14} aryl, particularly preferred C_1-C_6 alkyl, cyclohexyl or phenyl, particularly methyl or ethyl.

The solvent-containing gel in step c) is preferably reacted with a symmetric disiloxane, whereby a symmetric disiloxane is understood to be a disiloxane in which both Si atoms have the same residue R.

Particularly preferred, disiloxanes are used in which all residues R are the same. In particularly, one uses hexamethyldisiloxane.

The reaction is generally conducted at 20°C up to the boiling point of the silylation agent, if necessary, in a solvent. Preferred solvents here are the solvents described as preferred in step b). Particularly preferred is acetone, tetrahydrofuran, pentane and n-heptane. It the silylation is produced in a solvent,

then the silylation is generally conducted between 20°C and the boiling point of the solvent.

In a preferred form of embodiment, silylation is conducted in the presence of a catalyst, for example an acid or base. Preferably, acids are utilized as the catalyst. Particularly preferred acids are hydrochloric acid, sulfuric acid, acetic acid and/or phosphoric acid.

In another form of embodiment, the silylation is conducted in the presence of catalytic quantities of a silylation agent, which forms acids in the presence of water. Preferably, chlorosilanes are [used], and particularly preferred is trimethylchlorosilane (TMCS). In addition, a combination of acids or bases and TMCS is also possible.

Prior to step d), the silylated gel is preferably washed with a protic or aromatic solvent, until unreacted silylation agent is essentially removed (residual content ≤ 1 wt.%). Suitable solvents are those named in step b). Analogously, the solvents named there as preferred are also preferred here.

In step d), the silylated and, if necessary, washed gel is preferably dried subcritically, preferably at temperatures from -30°C to 200°C, and particularly preferred, 0 to 100°C, as well as pressures preferably from 0.001 to 20 bars, and particularly preferably 0.01 to 5 bars, particularly 0.1 to 2 bars, for example by radiation, convection and/or contact drying. Drying is preferably conducted until the gel has a residual solvent content of less than 1 wt.%. The aerogels obtained in the drying are permanently hydrophobic.

The gel obtained in step c) may also be dried sup rcritically. This requires temperatures higher than 200°C and/or pressures higher than 20 bars, depending on the respective solvent. This is possible without anything further, but it is associated with increased expenditure and does not offer essential advantages.

In another form of embodiment, the gel can be subjected to another network reinforcement, each time depending on application, prior to the silylation in step c). This is done by reacting the obtained gel with a solution of an orthosilicate of formula $R^1_{4-n}Si(OR^2)_n$ capable of condensation, preferably an alkyl and/or aryl orthosilicate, whereby n = 2 to 4 and R^1 and R^2, independently of one another, are hydrogen atoms, linear or branched C_1-C_6 alkyl, cyclohexyl or phenyl residues, or with an aqueous silicic-acid solution.

In another form of embodiment, after the shaping polycondensation and/or each subsequent process step, the gel can be comminuted according to techniques known to the person skilled in the art, such as, e.g., milling.

The aerogels produced according to the process of the invention find particular use as heat insulation materials.

The process according to the invention is described in more detail in the following, based on examples of embodiment, without thereby being limited to these.

Example 1

P7 ~~2 liters of a sodium water-glass solution (SiO_2 content of 6 wt.% and Na_2O:SiO_2 ratio of 1:3.3) are passed through a sheathed glass column (length =~~

100 cm, ~~diameter = 8 cm), which is packed with 4 liters of an acidic ion-~~ exchanger resin (styrene-divinylbenzene copolymer with sulfonic acid groups, commercially available under th name ®Duolite C20) (at approximately 70 ml/min). The column is operated at a temperature of approximately 7°C. The silicic-acid solution exiting at the lower end of the column has a pH value of 2.3. This solution is brought to a pH of 4.7 for the polycondensation with a 1.0 molar NaOH solution. After this, the gel that forms is aged for another 3 hours at 85°C and then the water is exchanged for acetone with 3 liters of acetone. Then the acetone-containing gel is silylated with hexamethyldisiloxane at room temperature for 5 hours (2.5 wt.% hexamethyldisiloxane per gram of wet gel). After washing the gel with 3 liters of acetone, drying of the gel is conducted in air (3 hours at 40°C, then 2 hours at 50°C and 12 hours at 150°C). The thus-obtained transparent aerogel has a density of 0.15 g/cm^3, a heat conductivity of 15 [16?] mW/mK, a specific surface according to BET of 600 m^2/g and is ~~permanently hydrophobic.~~

Example 2

424 g of a 7.5% HCl solution cooled to 10°C is reacted dropwise with 712 g of a sodium water-glass solution cooled to 10°C (with a content of 13 wt.% SiO_2 and a Na_2O:SiO_2 ratio of 1:3.3). A pH value of 4.7 is thereby adjusted. The hydrogel formed after several seconds is aged for one hour at 85°C. It is then washed with 3 liters of hot water and the water is exchanged for acetone with 3 liters of acetone. Then the acetone-containing gel is silylated with hexamethyldisiloxane (2.5 wt.% hexamethyldisiloxane per gram of wet gel) for 5

hours at room temperature. After washing the gel with 3 liters of acetone, it is dried in air (3 hours at 40°C, then 2 hours at 50°C and 12 hours at 150°C).

The thus-obtained aerogel has a density of 0.15 g/cm^3, a h at conductivity of 17 mW/mK, a specific surface according to BET of 580 m^2/g and is permanently hydrophobic.

Example 3

~~The hydrogel is produced as described in Example 2. The hydrogel aged~~ for one hour at 85°C is then washed with 3 liters of warm water and the water is exchanged for acetone with 3 liters of acetone. Then the acetone-containing gel is silylated with hexamethyldisiloxane (2.5 wt.% hexamethyldisiloxane per gram of wet gel) in the presence of 0.1 wt.% trimethylchlorosilane (0.? wt.% trimethylchlorosilane per gram of wet gel) for 5 hours at room temperature. After washing the gel with 3 liters of acetone, it is dried in air (3 hours at 40°C, then 2 ~~hours at 50°C and 12 hours at 150°C).~~

The thus-obtained aerogel has a density of 0.14 g/cm^3, a heat conductivity of 16 mW/mK, a specific surface according to BET of 590 m^2/g and is permanently hydrophobic.

Example 4

The hydrogel is produced as described in Example 2. The hydrogel aged for 1 hour at 85°C is then washed with 3 liters of warm water and the water is exchanged for acetone with 3 liters of acetone. Then the acetone-containing gel is silylated with hexamethyldisiloxane (2.5 wt.% hexamethyldisiloxane per gram

of wet gel) in the presence of 0.1 wt.% 1 N aqueous hydrochloric acid (0.1 wt.% 1 N aqueous hydrochloric acid per gram of wet gel) for 5 hours at room temperature. After washing the gel with 3 liters of acetone, it is dried in air (3 hours at 40°C, then 2 hours at 50°C and 12 hours at 150°C).

The thus-obtained aerogel has a density of 0.14 g/cm^3, a heat conductivity of 16 mW/mK, a specific BET surface of 570 m^2/g and is permanently hydrophobic.

The heat conductivities were measured with a resistance-wire method (see, e.g., O. Nielssen, G. Rüschenpöhler, J. Gross, J. Fricke, High Temperatures – High Pressures, Vol. 21, 267-274 (1989)).

[Patent Claims follow – previously translated]